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(54) **Method of producing a grain-oriented electrical steel sheet excellent in magnetic characteristics**

Verfahren zur Herstellung eines kornorientierten Elektrobleches mit ausgezeichneten magnetischen Eigenschaften

Procédé de fabrication d'une tôle d'acier à grains orientés presentant d'excellentes caracteristiques magnétiques

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- **PATENT ABSTRACTS OF JAPAN vol. 014, no. 456 (C-0765), 2 October 1990 (1990-10-02) & JP 02 182866 A (NIPPON STEEL CORP), 17 July 1990 (1990-07-17)**

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Description

[0001] This invention relates to a method of producing a grain-oriented electrical steel sheet excellent in magnetic characteristics for use primarily in the cores of electrical transformers and the like.

[0002] A generally known method used to produce a grain-oriented electrical steel sheet is to heat the slab to a very high temperature between 1350°C and a maximum of 1450°C and to hold (soak) the slab at this temperature for a period sufficient to ensure uniform heating throughout its entirety. This is for putting MnS, AlN and the like into solid solution so that they function as inhibitors. Since this method requires the slab to be heated to a very high temperature, however, it involves various problems in actual production. For example, (1) the slag produced by melting of the slab surface layer causes difficult problems from the point of heating furnace maintenance, (2) the desired hot-rolling temperature is difficult to secure, and (3) yield is reduced by large edge cracks occurring in hot-rolled strip.

[0003] Various technologies aimed at avoiding the requirement of such high temperature slab heating have been proposed. These are classified into two categories.

[0004] Those classified in the first category, such as taught by Japanese Unexamined Patent Publication Nos. Sho 59-56522 and Hei 5-112827 and Hei 9-118964 adopt a method that combines use of AlN as inhibitor, a slab heating temperature under 1280°C and nitriding up to the start of secondary recrystallization following decarburization annealing. As taught by JP-A-2-182866, for example, in order to ensure good secondary recrystallization in this method, it is extremely important to control the average diameter of primary recrystallization grains after decarburization annealing to within a prescribed range, usually to within the range of 18 ~ 35 μm. Since the method used for this control relies mainly on adjustment of the decarburization annealing temperature, however, the composition of the oxide layer after decarburization annealing is inevitably varied on top of the unavoidable variation in industrial production. This in turn may hinder formation of a glass film (containing spinel whose main component is forsterite) formed by reacting MgO as the main component with SiO_2 on the surface of the steel sheet. To prevent this hindrance from occurring, rigorous regulation of the conditions for glass film formation, including strict control of the MgO component, is required. In addition, the nitriding conditions become inconstant because of the variation in the composition of the oxide layer formed during decarburization and the resulting variation in the amount of nitrides formed may cause unstable secondary recrystallization. In particular, the method according to JP-A-9-118964, which uses a large amount of Mn in order to improve the iron loss of the steel sheet by increasing its resistivity, is not suitable for industrial production because it is very liable to produce defects in the glass film.

[0005] As taught, for example, by JP-A-6-322443, the technologies falling in the second category use Cu_xS (x = 1.8 or 2) as inhibitor and set the slab heating temperature to one not higher than the solution temperature of MnS and not lower than the solution temperature of Cu_xS. These methods are characterized by not requiring an additional process such as the nitriding in the first category of methods. However, there is nothing novel in using Cu_xS as an inhibitor for controlling secondary recrystallization and the method is not suitable for producing a high permeability grain-oriented electrical steel sheet having weak texture by applying a final cold rolling reduction ratio of greater than 80% (Iron and Steel, p. 2049, No. 15, Vol. 70, No. 1984). Specifically, as shown in Fig. 4, this technology, which does not conduct nitriding after decarburization annealing up to the start of secondary recrystallization during finish annealing and does not make effective use of AlN, cannot stably provide a product with a high magnetic flux density exceeding 1.89T on an industrial scale. Fig. 4 shows the relationship between the magnetic flux density (B_8 :T) and the iron loss ($W_{17/50}$: W/kg) of a thickness of 0.30 mm product. In Fig. 4, ☆, and ★ are examples when the heating rate in decarburization annealing was below 100°C/sec and above 100°C/sec, respectively, ◊ represents the TGO of JP-A-6-322443 and the broken line indicates the results of inventor tests and shows the secondary recrystallization to be poor. ○ indicates Comparative Examples of the present invention. In addition, although JP-A-6-322443 calls for precipitation of at least 60% of the total N content in the hot-rolled strip state as AlN, in the case of the hot-rolled strip having the composition shown in the Examples (Mn, S, Al, N) and obtained at a slab heating temperature on the level of 1270°C, uniform precipitation of AlN and MnS, which are precipitates whose solubility product is a quadratic function, is extremely difficult industrially. It is therefore impossible to obtain uniform magnetic characteristics throughout the coil length.

[0006] An object of this invention is to enable stable and simple production of a grain-oriented electrical steel sheet excellent in magnetic characteristics by compensating for the drawbacks of the technologies in both of the foregoing categories.

[0007] The present invention is defined by the appended claims.

[0008] The invention is described in more detail in connections with the drawings, in which:

Fig. 1 is a diagram showing the relationship between glass film defect ratio and Seq,

Fig. 2 is a diagram showing the relationship between magnetic property and nitriding level in the case of sheet thickness of 0.23 mm,

Fig. 3 is a diagram showing the relationship between magnetic property and nitriding level in the case of sheet thickness of 0.27 mm, and

Fig. 4 is a diagram showing the relationship between the magnetic flux density (B_8:T) and the iron loss ($W_{17/50}$:W/kg) of a thickness of 0.30 mm product. The open stars and solid stars are examples when the heating rate in decarburization annealing was below and above 100°C/sec, respectively.

[0009] The invention will now be explained in detail.

[0010] The most salient feature of this invention is that, in a method for producing a grain-oriented electrical steel sheet permitting a lower slab heating temperature than heretofore owing to avoidance of MnS as the main inhibitor for secondary recrystallization, it causes MnS (or MnSe), Cu_xS (or CuSe) etc. to function as primary inhibitors for controlling primary recrystallization grain diameter, causes nitrides (AlN, Si_3N_4 and individual or composite precipitates of Mn etc.) formed by nitriding conducted after decarburization annealing up to the start of secondary recrystallization in finish annealing to function as secondary inhibitors for controlling secondary recrystallization, and enables production of the grain-oriented electrical steel sheet excellent in magnetic characteristics by causing the two types of inhibitors to function effectively. In other words an object of the present invention is to metallurgically separate the functioning stages of inhibitors playing major roles in production of the grain-oriented electrical steel sheet and to cause each to fulfill its own function using different substances.

[0011] In production of the grain-oriented electrical steel sheet, the temperature of the decarburization annealing during which primary recrystallization takes place is generally low, i.e., no higher than 930°C. At this stage, therefore, the strong inhibitor formed during high-temperature hot rolling in the conventional method is unnecessary. Since the present invention uses sulfides and/or selenides as the primary inhibitors, the temperature dependence of the primary recrystallization grains is very small and, therefore, the temperature of the primary recrystallization annealing (actually the decarburization annealing) need not be greatly modified. This ensures very high stability of the primary oxide layer composition and the amount of nitrides formed by the ensuing nitriding, remarkably reduces glass film defects, and also eliminates nonuniformity of secondary recrystallization to enable stable industrial production.

[0012] On the other hand, in order to produce a sharp Goss orientation capable of providing good magnetic characteristics, the secondary recrystallization requires an inhibitor made stable against high temperatures by addition of sulfides and/or selenides. In the present invention, AlN formed by the nitriding mainly provides this stabilizing effect.

[0013] The reasons for limiting the content ranges of the slab components will now be explained.

[0014] When the C content is under 0.025%, the primary recrystallization texture becomes inappropriate, and when it is over 0.10%, the steel sheet becomes inappropriate for industrial production owing to difficulty of decarburization.

[0015] When the Si content is under 2.5%, desired iron loss property cannot be obtained, and when it is over 4.0%, the steel sheet becomes inappropriate for industrial production owing to extreme difficulty of cold rolling.

[0016] When the Mn content is under 0.02%, yield decreases because the hot-rolled strip becomes susceptible to cracking. When the Mn content is over 0.20%, problems arise regarding production stability in actual industrial production because the amount of MnS and/or MnSe increases to the point of causing local differences in the degree of their solid solution state and their amount. S and Se combine with Mn and Cu to form mainly primary inhibitors. When Seq which is a sulfur equivalent value represented by (S+0.406Se) is under 0.008%, however, the primary inhibitor strength becomes too weak to control the primary recrystallization. In this case, the variation in primary recrystallization grain diameter with decarburization annealing temperature becomes so large as to require change of the decarburization annealing temperature depending on the composition. On the other hand, when Seq is over 0.050%, the inhibitor strength becomes too strong, so that poor secondary recrystallization occurs owing to insufficient primary recrystallization.

[0017] Al combines with N to form AlN, which functions mainly as a secondary inhibitor. Some AlN is formed before nitriding and some is formed during the high-temperature annealing after nitriding. An Al content of 0.0010 ~ 0.035% is needed to ensure the required amounts of AlN both before and after nitriding. When the Al content is outside this range, the primary recrystallization grain diameter becomes difficult to control and the secondary recrystallization therefore does not proceed stably.

[0018] As pointed out in the foregoing, the present invention uses mainly sulfides and selenides to control the primary recrystallization grains. However, AlN contained in the slab is also necessary for primary recrystallization grain control, and control of the primary recrystallization grain diameter is difficult when the N content is below 0.0030%. On the other hand, the upper limit of the N content is defined as 0.010% because at higher contents defects, i.e., blisters, occur on the steel sheet surface. Owing to this limitation, the amount of N contained in the slab is not sufficient to control the secondary recrystallization. This is why the nitriding explained later is necessary.

[0019] When the slab is heated to 1050°C or higher and hot-rolled under the present invention conditions, Cu is finely precipitated together with S and Se and manifests a primary inhibitor effect. As the precipitates also act as precipitation nuclei that more evenly disperse the AlN, they also play a role as secondary inhibitor and by this effect improve the secondary recrystallization. When Cu is present at under 0.02%, these effects decrease to the point of making stable production difficult, while presence of Cu at over 0.3% provides little or no additional effect and causes surface defects called "copper spills (Scab)" during hot rolling

[0020] Sn, Sb and P contribute to improvement of the primary recrystallization texture. Cr has a beneficial effect on formation of a forsterite film (glass film). When the contents of these elements are below the ranges set out above, the beneficial effects on formation of a forsterite film are slight. When they are above the stated ranges, it becomes difficult to form a stable forsterite film (glass film). As Ni is highly effective for obtaining uniform dispersion of precipitates as primary and secondary inhibitors, its addition further improves and stabilizes the magnetic characteristics. It has no effect when added to less than 0.02%, while addition to over 0.3% makes formation of a forsterite film difficult because it impedes oxygen enrichment after decarburization annealing.

[0021] Mo and Cd also contribute to inhibitor strengthening by forming sulfides and selenides. They have no effect at a content under 0.008%, while when present at over 0.3%, they cause formation of enlarged precipitates that do not function as inhibitors that stabilize the magnetic characteristics.

[0022] The reasons for limiting the production steps in the present invention will now be explained.

[0023] Although the average diameter of the primary recrystallization grains after completion of decarburization annealing is specified as 18 ~ 35 μm in Japanese Patent Application 06-046161, for example, in the present invention the average grain diameter of the primary recrystallization grains must be not less than 7 μm and less than 15 μm. This is an extremely important point of the invention as regards achieving excellent magnetic characteristics (particularly iron loss property). One reason for this is that, from the viewpoint of grain growth, the volume fraction of Goss oriented grains that can grow as secondary recrystallization grains becomes greater at the primary recrystallization stage when the primary recrystallization grain diameter is smaller (Materials Science Forum, Vol. 204 ~ 206, Part 2, p. 631). Another is that the number of Goss nuclei becomes greater owing to the small grain diameter. Since the absolute number of Goss nuclei is therefore several times greater in this invention than when the average diameter of the primary recrystallization grains is 18 ~ 35 μm, the secondary recrystallization grain diameter is also relatively smaller and the iron loss property is proportionally better.

[0024] Since, moreover, the small average diameter of the primary recrystallization grains increases the driving force of the secondary recrystallization, the secondary recrystallization starts early at the temperature of heating stage (at a low-temperature point) of the final finish annealing. In light of the current practice of final finish-annealing sheet in the coiled state, this means that the temperature hysteresis at different points of the coil up to the maximum temperature becomes more uniform (that the temperature increase rate becomes constant). As nonuniformity among different coil locations is therefore markedly reduced, the magnetic characteristics are highly stable.

[0025] The present invention requires that the steel sheet be nitrided between the completion of the decarburization annealing and the start of secondary recrystallization. This can be achieved either by the method of mixing nitrides (CrN, MnN and the like) with the annealing separator used during high-temperature annealing or by the method of nitriding the decarburization-annealed sheet as a running strip in an atmosphere containing ammonia. While either method can be used, the latter exhibits better stability in industrial production. When the amount of nitridation is below 0.001%, the secondary recrystallization is unstable, and when it is over 0.020%, many defects exposing the matrix occur in the glass film. The preferred range is 0.005 ~ 0.015%.

[0026] The slab heating temperature prior to hot rolling is an important factor in this invention. Ultra-high temperature slab heating to a temperature exceeding 1350°C encounters severe difficulties in industrial production. Below the lower limit of 1050°C, on the other hand, the hot rolling becomes practically difficult and, moreover, the generation of primary inhibitor, a key point of the present invention, falls to an insufficient level that causes the primary recrystallization grain diameter to vary greatly with the decarburization annealing temperature. From the viewpoint of ease of hot rolling and the shape (crown) of the hot-rolled strip, the preferred slab heating temperature range is 1200 ~ 1300°C.

[0027] The hot rolling temperature is, moreover, prescribed as:

$$850 + 2500 \times Seq + 400 \times Mn \leqq \text{FOT (starting temperature of finishing hot-rolling)} \leqq 1100 + 3000 \times Seq + 800 \times Mn \leqq 1350^\circ C$$

$$800 + 2500 \times Seq + 400 \times Mn \leqq \text{FT(finishing temperature of finishing hot-rolling)} \leqq 1050 + 3000 \times Seq + 800 \times Mn \leqq 1350^\circ C.$$

[0028] Below these ranges, sulfides and selenides precipitate excessively and fail to function as primary inhibitors. Since the primary recrystallization grain diameter therefore becomes highly dependent on decarburization annealing temperature, control becomes difficult in industrial production. Hot rolling temperatures above these ranges are not suitable in industrial production because, in actual production, the amount and state of MnS (MnSe) about solid solution comes to vary between different locations in the material, thereby causing local variation in secondary recrystallization.

[0029] In the present invention method, a slab is first produced by the conventional continuous casting method to have an initial thickness in the range of 150 mm to 300 mm, preferably 200 mm to 250 mm. It is also possible instead to use a so-called thin slab with an initial thickness in the range between about 30 mm and 70 mm. These ranges are advantageous in that no roughing rolling down to an intermediate thickness is needed at the time of producing the hot-rolled strip. If a slab or strip is produced beforehand by strip casting, moreover, a grain-oriented electrical steel sheet can be produced by the invention using a slab or strip having an even thinner initial thickness.

[0030] The heating method adopted for the hot rolling in industrial production is not limited to ordinary gas heating but can instead be induction heating or direct electric heating. No problem is encountered when the shape needed for these special heating methods is obtained by effecting breakdown on the cast slab. When the heating temperature is high, i.e., over 1300°C, this breakdown can be used to improve the texture and lower the amount of C. These are known techniques in the art.

[0031] In the cold rolling, when the final cold rolling reduction ratio is under 80%, the {110}<001> texture broadens to the point of making it impossible to obtain high magnetic flux density. On the other hand, a final cold rolling reduction ratio of over 92% reduces the {110}<001> texture to such an extremely low level that the secondary recrystallization becomes unstable.

[0032] The annealing of the hot-rolled strip is conducted mainly for the purpose of eliminating the nonuniformity of the texture/inhibitor dispersion that occurs in the strip during hot rolling. The annealing can be effected on the hot-rolled strip or be effected prior to the final cold rolling. At least one continuous annealing is preferably conducted before the final cold rolling in order to even out the heat hysteresis that arises during hot rolling.

[0033] Although the final cold rolling can be conducted at normal temperature, holding the strip the temperature range of 100 ~ 300°C for at least one minute during at least one final cold rolling pass improves the primary recrystallization texture and markedly enhances the magnetic characteristics.

[0034] Making the heating rate between room temperature and 650 ~ 950°C in the decarburization annealing not less than 100°C/sec improves the primary recrystallization texture and enhances the magnetic characteristics. Various methods are available for securing the heating rate. These include resistance heating, induction heating, and direct energy transfer heating. It is known from JP-B-(examined published Japanese patent application) 6-51887, among others, that speeding up the heating rate increases the Goss orientation in the primary recrystallization texture and reduces the secondary recrystallization grain diameter. While JP-B-6-51887 specifices a heating rate of not less than 40°C/sec, in the present invention the heating rate is effective even at 100°C/sec and is preferably 150°C/sec or higher. The decarburization annealing temperature is specified as not lower than 650°C because the effect is low below this temperature owing to incomplete recrystallization, and is specified as not higher than 950°C because decarburization annealing temperatures in excess of 950°C are not used in the production of grain-oriented electrical steel sheet.

<Example 1>

[0035] Table 1 shows the composition of molten steels produced by an ordinary method, and Table 2 shows the production conditions and the resulting product characteristics. Continuous annealing was conducted at 1100°C for 150 seconds followed by cooling at 20°C/sec. Annealing was then conducted at 850°C for 90 ~ 150 seconds in a mixed atmosphere of H_2 and N_2 having a dew point of 65°C. This decarburization annealing was conducted at different heating rates of 50°C/sec, 110°C/sec and 180°C/sec. Nitriding by the designated method before and after coating with an annealing separator composed mainly of MgO and secondary recrystallization annealing were then conducted. The secondary recrystallization annealing was carried out in an atmosphere of N_2 = 25%, H_2 = 75% at 10 ~ 20°C/hr up to 1200°C. Following this purification annealing was conducted at a temperature of 1200°C for 20 or more hours in an atmosphere of H_2 = 100%. Application of an ordinarily used insulating tension coating and flattening annealing were then conducted.

Table 1

No		Molten steel composition wt%															
		C	Si	S	Se	Seq	Al	N	Mn	Cu	Sn	Sb	Ni	P	Cr	Mo	Cd
1	Invention	0.054	3.28	0.022	--	0.022	0.024	0.0078	0.08	0.15	0.08	--	--	0.02	--	--	--
2	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
3	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
4	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
5	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
6	Invention	0.054	3.28	0.023	--	0.023	0.024	0.0078	0.10	0.15	0.08	--	--	0.02	0.10	--	--
7	Invention	0.054	3.28	0.023	--	0.023	0.024	0.0078	0.10	0.15	0.08	--	--	0.02	0.10	--	--
8	Invention	0.054	3.28	0.023	--	0.023	0.024	0.0078	0.10	0.15	0.08	--	--	0.02	0.10	--	--
9	Comparison	0.051	3.23	0.006	--	0.006	0.023	0.0080	0.07	0.15	0.08	--	--	0.03	--	--	--
10	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
11	Comparison	0.054	3.28	0.053	--	0.053	0.025	0.0078	0.05	0.15	--	0.08	--	0.02	--	--	--
12	Invention	0.055	3.35	0.024	--	0.024	0.025	0.0082	0.05	0.15	0.08	--	--	0.02	--	--	--
13	Invention	0.055	3.35	0.024	--	0.024	0.025	0.0082	0.05	0.15	0.08	--	--	0.02	--	--	--
14	Invention	0.055	3.35	0.024	--	0.024	0.025	0.0082	0.05	0.15	0.08	--	--	0.02	--	--	--
15	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--

Table 1 (Continued)

No		Molten steel composition wt%															
		C	Si	S	Se	Seq	Al	N	Mn	Cu	Sn	Sb	Ni	P	Cr	Mo	Cd
16	Invention	0.050	3.37	0.023	--	0.023	0.024	0.0085	0.09	0.13	0.05	--	0.08	0.02	0.09	--	--
17	Invention	0.050	3.37	0.023	--	0.023	0.024	0.0085	0.09	0.13	0.05	--	0.08	0.02	0.09	--	--
18	Invention	0.050	3.37	0.023	--	0.023	0.024	0.0085	0.09	0.13	0.05	--	0.08	0.02	0.09	--	--
19	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
20	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
21	Invention	0.049	3.20	0.007	0.055	0.029	0.020	0.0075	0.14	0.08	--	--	0.08	0.02	0.09	--	--
22	Invention	0.049	3.20	0.007	0.055	0.029	0.020	0.0075	0.14	0.08	--	--	0.08	0.02	0.09	--	--
23	Invention	0.049	3.20	0.007	0.055	0.029	0.020	0.0075	0.14	0.08	--	--	0.08	0.02	0.09	--	--
24	Comparison	0.049	3.20	0.007	0.055	0.029	0.020	0.0075	0.14	0.08	--	--	0.08	0.02	0.09	--	--
25	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
26	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
27	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
28	Comparison	0.058	3.13	0.007	--	0.007	0.028	0.0075	0.15	--	--	--	--	--	--	--	--
29	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
30	Invention	0.060	3.25	0.011	"	0.011	0.026	0.0061	0.07	0.20	0.12	"	"	0.05	"	--	--

Table 1 (Continued)

No		Molten steel composition wt%															
		C	Si	S	Se	Seq	Al	N	Mn	Cu	Sn	Sb	Ni	P	Cr	Mo	Cd
31	Invention	0.060	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
32	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
33	Invention	0.060	3.25	0.011	"	0.011	0.026	0.0061	0.07	0.20	0.12	"	"	0.05	"	--	--
34	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
35	Invention	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
36	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
37	Comparison	"	"	"	"	"	"	"	"	"	"	"	"	"	"	--	--
38	Invention	0.064	3.35	0.015	--	0.015	0.026	0.0070	0.07	0.10	0.12	--	--	0.02	--	0.01	--
39	Invention	"	"	"	"	"	"	"	"	"	"	--	"	"	--	"	0.05
40	Invention	"	"	"	"	"	"	"	"	"	"	--	0.07	0.05	--	"	"
41	Comparison	"	"	"	"	"	"	"	"	"	"	--	"	"	--	"	0.10
42	Comparison	"	"	"	"	"	"	"	"	"	"	--	"	"	--	"	"

Table 2

No		Slab heating temp	Continuous annealing or not: hot rolled thickness	Continuous annealing or not: Intermediate thickness	Final cold rolling reduction ratio	Final product thickness	Decarburization annealing heating rate	Average primary recrystallization grain diameter	Nitriding: Method	Nitriding: Amount	Magnetic property: $W_{17/50}$	Magnetic property: B_8	Glass film defect rate
		°C	Thickness mm		%	mm	°C/sec	µm		%	W/kg	T	%
1	Invention	1300	2.3	--	87.0	0.30	50	13.2	A	0.012	0.95	1.94	1.5
2	Invention	"	"	--	"	"	110	13.0	A	0.012	0.92	1.94	1.4
3	Invention	"	"	--	"	"	180	12.3	A	0.012	0.90	1.93	1.5
4	Comparison	"	"	"	"	"	50	13.2	-	--	Poor secondary recrystallizatiion		0.5
5	Comparison	"	2.1	"	83.3	0.35	50	13.5	-	--	1.25	1.84	0.4
6	Invention	1300	2.3	--	87.0	0.30	50	13.3	A	0.012	0.96	1.95	0.5
7	Invention	1300	2.3	--	87.0	0.30	110	12.9	A	0.012	0.94	1.94	0.6
8	Invention	1300	2.3	--	87.0	0.30	180	12.5	A	0.012	0.91	1.93	0.5
9	Comparison	1250	2.3	--	87.0	0.30	50	15.8	A	0.013	1.18	1.83	1.5
10	Comparison	1375	2.3	--	87.0	0.30	50	6.5	A	0.012	1.21	1.81	1.5
11	Comparison	1250	2.3	--	87.0	0.30	50	6.0	A	0.012	1.30	1.80	1.5

Table 1 (Continued)

No		Slab heating temp	Continuous annealing or not: hot rolled thickness	Continuous annealing or not: Intermediate thickness	Final cold rolling reduction ratio	Final product thickness	Decarburization annealing heating rate	Average primary recrystallization grain diameter	Nitriding: Method	Nitriding: Amount	Magnetic property $W_{17/50}$	Magnetic property B_8	Glass film defect rate
			Thickness										
		°C	mm	mm	%	mm	°C/sec	µm		%	W/kg	T	%
12	Invention	1270	2.3	1.55	85.2	0.23	50	14.0	A	0.004	0.84	1.94	1.7
13	Invention	1270	2.3	1.55	85.2	0.23	110	13.5	A	0.012	0.80	1.93	1.6
14	Invention	1270	2.3	1.55	85.2	0.23	180	13.1	A	0.012	0.78	1.93	1.5
15	Comparison	1270	2.3	1.55	85.2	0.23	50	14.0	-	--	Poor secondary recrystallizatiion		0.7
16	Invention	1280	2.3	1.55	85.2	0.23	50	12.8	A	0.012	0.81	1.94	0.9
17	Invention	1280	2.3	1.55	85.2	0.23	110	11.9	A	0.012	0.79	1.95	0.8
18	Invention	1280	2.3	1.55	85.2	0.23	180	11.2	A	0.012	0.76	1.94	0.9
19	Comparison	1280	2.3	1.55	85.2	0.23	50	13.5	-	--	1.05	1.83	0.5
20	Comparison	1285	3.0	--	92.3	0.23	50	12.2	A	0.011	Poor secondary recrystallizatiion		1.1
21	Invention	1260	2.3	1.55	85.2	0.23	50	12.5	A	0.012	0.80	1.93	0.8

Table 2 (Continued)

No		Slab heating temp	Continuous annealing or not: hot rolled thickness (Thickness)	Continuous annealing or not: Intermediate thickness (Thickness)	Final cold rolling reduction ratio	Final product thickness	Decarburization annealing heating rate	Average primary recrystallization grain diameter	Nitriding: Method	Nitriding: Amount	Magnetic property $W_{17/50}$	Magnetic property B_8	Glass film defect rate
		°C	mm	mm	%	mm	°C/sec	µm		%	W/kg	T	%
22	Invention	1280	2.3	1.55	85.2	0.23	110	12.1	A	0.012	0.79	1.94	0.9
23	Invention	1280	2.3	1.55	85.2	0.23	180	11.8	A	0.012	0.77	1.92	0.8
24	Comparison	1280	2.3	1.55	85.2	0.23	50	12.5	-	--	Poor secondary recrystallization		0.4
25	Invention	"	2.3	--	90.0	0.23	50	11.9	A	0.013	0.79	1.95	0.9
26	Invention	"	2.3	--	90.0	0.23	110	11.3	A	0.013	0.77	1.95	0.8
27	Invention	"	2.3	--	90.0	0.23	180	10.8	A	0.013	0.75	1.94	1.0
28	Comparison	"	2.3	--	90.0	0.23	50	19.0	-	--	Poor secondary recrystallization		0.9
29	Comparison	"	"	--	90.0	0.23	50	19.0	B	0.025	0.94	1.95	13.5
30	Invention	1270	"	--	90.0	0.23	50	13.8	B	0.010	0.83	1.94	2.5
31	Invention	"	"	--	90.0	0.23	110	13.4	B	0.010	0.81	1.93	2.8

Table 2 (Continued)

No		Slab heating temp	Continuous annealing or not: hot rolled thickness (Thickness)	Continuous annealing or not: Intermediate thickness (Thickness)	Final cold rolling reduction ratio	Final product thickness	Decarburization annealing heating rate	Average primary recrystallization grain diameter	Nitriding: Method	Nitriding: Amount	Magnetic property $W_{17/50}$	Magnetic property B_8	Glsss film defect rate
		°C	mm.		%	mm	°C/sec	µm		%	W/kg	T	%
32	Invention	"	"	--	90.0	0.23	180	12.9	B	0.009	0.79	1.94	2.5
33	Invention	1270	"	--	90.0	0.23	50	13.8	A	0.010	0.82	1.94	1.5
34	Invention	"	"	--	90.0	0.23	110	13.4	A	0.010	0.80	1.93	0.8
35	Invention	"	"	--	90.0	0.23	180	12.9	A	0.009	0.78	1.93	1.5
36	Comparison	"	"	--	90.0	0.23	180	12.9	-	--	Poor secondary recrystallizatiion		1.0
37	Comparison	"	"	--	88.5	0.23	180	17.0	-	--	Poor secondary recrystallizatiion		1.0
38	Invention	1300	2.3	--	87.0	0.30	50	14.2	A	0.011	0.94	1.95	1.6
39	Invention	"	"	--	"	"	110	15.0	A	0.010	0.93	1.96	1.3
40	Invention	"	"	--	"	"	180	14.3	A	0.011	0.88	1.95	1.2

Table 2 (Continued)

No		Slab heating temp	Continuous annealing or not: hot rolled thickness	Continuous annealing or not: Intermediate thickness	Final cold rolling reduction ratio	Final product thickness	Decarburization annealing heating rate	Average primary recrystallization grain diameter	Nitriding: Method	Nitriding: Amount	Magnetic property: $W_{17/50}$	Magnetic property: B_8	Glass film defect rate
		°C	Thickness mm	Thickness mm	%	mm	°C/sec	µm		%	W/kg	T	%
41	Comparison	″	″	--	″	″	50	14.4	-	--	Poor secondary recrystallization		0.7
42	Comparison	″	2.1	--	83.3	0.35	50	15.4	-	--	1.27	1.80	0.6

Nitriding method: A: Nitriding of running strip
B: Addition of MnN to annealing separator

<Example 2>

[0036] Fig. 1 shows the glass film defect rate when production was carried out under the following conditions using a material having the compositions set out below. Slabs comprising 0.045 ~ 0.065% of C, 3.0 ~ 3.51% of Si, 0.05 ~ 0.10% of Mn, 0.0060 ~ 0.0087% of N, 0.08 ~ 0.20% of Cu, 0.020 ~ 0.030% of Al, and amounts of Se and S selected to make Seq = 0.006 ~ 0.050% were produced. Each was heated to 1200 ~ 1300°C, formed into a 2.3 mm-thick hot-rolled strip, subjected to hot-rolled strip annealing at 980°C for 120 sec, pickled, cold-rolled to a sheet thickness of 1.55 mm, annealed at 1100°C for 150 sec, and final cold-rolled to a sheet thickness of 0.23 mm. At this time, the sheet was held at 180 ~ 220°C for not less than 2 min in at least two passes. Then, in order to obtain an average primary recrystallization grain diameter of 13 ~ 15 μm, the sheet was annealed in an atmosphere of H_2 = 75%, N_2 = 25% and dew point of 62°C at, in case (1) of Seq < 0.008%, an annealing temperature of 820 ~ 870°C for 90 seconds, and at, in case (2) of Seq ≧ 0.008%, an annealing temperature of 850°C for 90 sec, whereafter 0.008 ~ 0.012% nitriding was effected on a running strip in an ammonia atmosphere, an annealing separator composed mainly of MgO was applied, and high-temperature annealing was conducted. The high-temperature annealing was conducted to 1200°C in H_2 = 75%, N_2 = 25% at a temperature increase rate of 15°C/hr. Purification annealing was then conducted at 1200°C for 25 hours in H_2 = 100%. This was followed by application of an ordinarily used insulating tensile coating and flattening annealing.

[0037] As shown in Fig. 1, when Seq < 0.008%, the decarburization annealing temperature has to be changed to obtain primary recrystallization grains of uniform diameter, the oxide layer is not constant, and the glass film defect rate varies and is poor in absolute value. In contrast, when Seq ≧ 0.008%, the decarburization annealing temperature can be constant, the oxide layer is substantially constant, and the glass film defect rate is good and stable.

[0038] In passing it is noted that secondary recrystallization was good when Seq ≦ 0.050%.

<Example 3>

[0039] Production was carried out under the following conditions using a material having the compositions set out below. Figs. 2 and 3 show how the magnetic characteristics differed depending on whether or not nitriding was conducted in the case of sheets of 0.23 mm and 0.27 mm thickness. Slabs comprising 0.045 ~ 0.065% of C, 3.00 ~ 3.51% of Si, 0.05 ~ 0.10% of Mn, 0.0060 ~ 0.0087% of N, 0.08 ~ 0.20% of Cu, 0.020 ~ 0.030% of Al, and amounts of Se and S selected to make Seq = 0.010 ~ 0.025% were produced. Each was heated to 1200 ~ 1300°C, formed into a 2.3 mm-thick hot-rolled strip. In the case (1) where the final cold-rolled thickness was to be 0.23 mm, the strip was subjected to hot-rolled strip annealing at 980°C for 120 seconds, pickled, cold-rolled to a sheet thickness of 1.55 mm, annealed at 1100°C for 150 seconds, and final cold-rolled to the sheet thickness of 0.23 mm. In this case, the sheet was held at 180 ~ 220°C for not less than 2 minutes in at least two passes. It was then annealed in an atmosphere of H_2 = 75%, N_2 = 25% and dew point of 62°C at an annealing temperature of 850°C for 90 seconds. In the case (2) where the final cold-rolled thickness was to be 0.27 mm, the strip was subjected to hot-rolled strip annealing at 1120°C for 120 seconds, pickled, and final cold-rolled to the sheet thickness of 0.27 mm. In this case, the sheet was held at 180 ~ 220°C for not less than 2 minutes in at least two passes. It was then annealed in an atmosphere of H_2 = 75%, N_2 = 25% and dew point of 62°C at an annealing temperature of 850°C for 120 seconds.

[0040] Both nitrided (0.005 ~ 0.013% nitriding on a running strip in an ammonia atmosphere) and un-nitrided 0.23 mm sheets and 0.27 mm sheets were produced. Each was applied with an annealing separator composed mainly of MgO and high-temperature annealed. The high-temperature annealing was conducted to 1200°C at a temperature increase rate of 15°C/hr in (1) H_2 = 75%, N_2 = 25%, (2) H_2 = 50%, N_2 = 50%, (3) H_2 = 25%, N_2 = 75% and (4) H_2 = 10%, N_2 = 90%. Purification annealing was then conducted at 1200°C for 25 hours in H_2 = 100%. This was followed by application of an ordinarily used insulating tensile coating and flattening annealing. The results are shown in Figs. 2 and 3. As can be seen in Figs. 2 and 3, irrespective of which of the atmospheres (1) - (4) the nitriding was conducted in at the time of the high-temperature annealing temperature rise, both the 0.23 mm sheets and the 0.27 mm sheets exhibited excellent magnetic characteristics while the sheets that were not nitrided exhibited inferior magnetic characteristics despite secondary recrystallization.

[0041] Since application of the production method of this invention sharply reduces occurrence of glass film defects and eliminates nonuniformity of secondary recrystallization, stable industrial production becomes possible and excellent magnetic characteristics can be secured.

Claims

1. A method for producing a grain-oriented electrical steel containing 2.5 ~ 4.0% of Si, wherein at least one member selected from among sulfides and selenides is used as a first inhibitor and at least one nitride formed by nitriding

up to the start of secondary recrystallization following decarburization annealing is used as a second inhibitor, wherein the slab heating temperature prior to hot rolling is between 1260°C and 1350°C and primary recrystallization grains after completion of the decarburization annealing have a average grain diameter of not less than 7 μm and not larger than 15 μm.

2. A method for producing a grain-oriented electrical steel sheet according to claim 1 comprising the steps of reheating to a temperature in the range of 1260°C to 1350°C a slab comprising, by weight%, 0.025 ~ 0.10% of C, 2.5 ~ 4.0% of Si, 0.010 ~ 0.035% of acid-soluble Al, 0.0030 ~ 0.010% of N, Seq=(S + 0.406 Se)

of 0.008 ~ 0.05%, 0.02 ~ 0.20% of Mn , optionally at least one selected from 0.02~0.30% of at least one of Sn, Sb and P, 0.01~0.30% of Cu, 0.02~0.30% of Cr, 0.03~ 0.30% of Ni and 0.008~0.3% of at least one of Mo and Cd and the balance

being Fe and unavoidable impurities, hot-rolling the slab into a hot-rolled strip, optionally annealing the hot-rolled strip, subjecting to one cold rolling or to two or more cold rollings the hot-rolled strip with intermediate annealing to form a final sheet thickness,

decarburization annealing the cold-rolled sheet, coating the sheet with an annealing separator composed mainly of MgO and subjecting to final finish annealing, wherein the method is **characterized by** further comprising the step of nitriding the steel sheet from after the decarburization annealing up to the start of secondary recrystallization, and primary recrystallization grains after completion of the decarburization annealing have a average grain diameter of not less than 7 μm and not larger than 15 μm.

3. A method for producing a grain-oriented electrical steel containing 2.5 ~ 4.0% of Si, wherein at least one member selected from among sulfides and selenides is used as a first inhibitor and at least one nitride formed by nitriding up to the start of secondary recrystallization following decarburization annealing is used as a second inhibitor, wherein the initial thickness of a slab for the grain-oriented electrical steel sheet is in the range between about 30 mm and 70 mm and primary recrystallization grains after completion of the decarburization annealing have an average grain diameter of not less than 7 μm and not larger than 15 μm.

4. A method for producing a grain-oriented electrical steel sheet according to claim 3 comprising the steps of reheating to a temperature of more than 1050°C and lower than 1350°C a slab having an initial thickness of about 30 to 70 mm comprising, by weight%, 0.025 ~ 0.10% of C, 2.5 ~ 4.0% of Si, 0.010 ~ 0.035% of acid-soluble Al, 0.0030 ~ 0.010% of N, Seq=(S + 0.406 Se)

of 0.008 ~ 0.05%, 0.02 ~ 0.20% of Mn, optionally at least one selected from 0.02~0.30% of at least one of Sn, Sb and P, 0.01~0.30% of Cu, 0.02~0.30% of Cr, 0.03~ 0.30% of Ni and 0.008~0.3% of at least one of Mo and Cd and the balance

being Fe and unavoidable impurities, hot-rolling the slab into a hot-rolled strip, optionally annealing the hot-rolled strip, subjecting to one cold rolling or to two or more cold rollings the hot-rolled strip with intermediate annealing to form a final sheet thickness, decarburization annealing the cold-rolled sheet, coating the sheet with an annealing separator composed mainly of MgO and subjecting to final finish annealing, wherein the method is **characterized by** further comprising the step of nitriding the steel sheet from after the decarburization annealing up to the start of secondary recrystallization, and primary recrystallization grains after completion of the decarburization annealing have an average grain diameter of not less than 7 μm and not larger than 15 μm.

5. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to any of claims 1 to 4, wherein the step of nitriding the steel sheet after decarburization annealing is effected on a running strip in a mixed gas atmosphere containing hydrogen, nitrogen and ammonia to increase the amount of nitrogen in the steel sheet by 0.001 ~ 0.020 wt%.

6. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to any of claims 1 to 5, wherein heating during the decarburization annealing is conducted at a heating rate of not less than 100°C/sec from start of temperature rise to 650 ~ 950°C.

7. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to any of claims 1 to 6,
wherein the strip temperature is adjusted to temperatures within the following ranges during the hot rolling.

$$850 + 2500 \times Seq + 400 \times Mn \leqq FOT(^\circ C) \leqq 1100 +$$

$$3000 \times Seq + 800 \times Mn \leqq 1350°C$$

where FOT:starting temperature of finishing hot-rolling (°C),

$$800 + 2500 \times Seq + 400 \times Mn \leqq FT(°C) \leqq 1050 + 3000 \times Seq + 800 \times Mn \leqq 1350°C$$

where FT: finishing temperature of finishing hot-rolling (°C).

8. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to any of claims 1 to 7, wherein the hot-rolled strip annealing conditions are set to a maximum temperature of 950 ~ 1150°C and an annealing period of not less than 30 seconds and not more than 600 seconds.

9. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to any of claims 1 to 8, wherein the cold rolling is conducted at a final cold rolling reduction ratio of 80 ~ 92%.

10. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to any of claims 1 to 9, wherein the strip is held in the temperature range of 100 ~ 300°C for at least 1 min during at least one final cold rolling pass of the cold rolling.

11. A method for producing a grain-oriented electrical steel sheet excellent in magnetic characteristics according to claim 1 or 3, wherein at least one selected from 0.02~0.30% of at least one of Sn, Sb and P, 0.01~0.30% of Cu, 0.02~0.30% of Cr, 0.03~ 0.30% of Ni and 0.008~0.3% of at least one of Mo and Cd is further included as a component of the slab.

Patentansprüche

1. Verfahren zur Herstellung eines Kornorientierten Elektrostahlblechs, das 2,5 bis 4,0 % Si aufweist, wobei mindestens ein Bestandteil, der aus Sulfiden und Seleniden ausgewählt ist, als erster Inhibitor verwendet wird und mindestens ein Nitrid, das durch Nitrieren bis zum Beginn der sekundären Rekristallisation nach Entkohlungsglühen gebildet wird, als zweiter Inhibitor verwendet wird, wobei die Brammenerwärmungstemperatur vor Warmwalzen zwischen 1260 °C und 1350 °C liegt und primäre Rekristallisationskörner nach Abschluß des Entkohlungsglühens einen mittleren Korndurchmesser von mindestens 7 µm und höchstens 15 µm haben.

2. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs nach Anspruch 1 mit den folgenden Schritten: auf eine Temperatur im Bereich von 1260 °C bis 1350 °C erfolgendes Nachwärmen einer Bramme, die in Gew.-% aufweist: 0,025 bis 0,10 % C, 2,5 bis 4,0 % Si, 0,010 bis 0,035 % säurelösliches Al, 0,0030 bis 0,010 % N, Seq = (S + 0,406 Se) von 0,008 bis 0,05 %, 0,02 bis 0,20 % Mn, optional mindestens einen Bestandteil, ausgewählt aus 0,02 bis 0,30 % Sn, Sb und/oder P, 0,01 bis 0,30 % Cu, 0,02 bis 0,30 % Cr, 0,03 bis 0,30 % Ni und 0,008 bis 0,3 % Mo und/oder Cd und als Rest Eisen sowie unvermeidliche Verunreinigungen, Warmwalzen der Bramme zu einem warmgewalzten Band, optionales Glühen des warmgewalzten Bands, einmaliges Kaltwalzen oder zweioder mehrmaliges Kaltwalzen des warmgewalzten Bands mit Zwischenglühen, um eine Fertigblechdicke zu bilden, Entkohlungsglühen des kaltgewalzten Blechs, Beschichten des Blechs mit einem sich hauptsächlich aus MgO zusammensetzenden Glühseparator und abschließendes Fertigglühen, wobei das Verfahren **dadurch gekennzeichnet ist, daß** es ferner den Schritt des Nitrierens des Stahlblechs nach dem Entkohlungsglühen und bis zum Beginn der sekundären Rekristallisation aufweist und primäre Rekristallisationskörner nach Abschluß des Entkohlungsglühens einen mittleren Korndurchmesser von mindestens 7 µm und höchstens 15 µm haben.

3. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs, das 2,5 bis 4,0 % Si aufweist, wobei mindestens ein Bestandteil, der aus Sulfiden und Seleniden ausgewählt ist, als erster Inhibitor verwendet wird und mindestens ein Nitrid, das durch Nitrieren bis zum Beginn der sekundären Rekristallisation nach Entkohlungsglühen gebildet wird, als zweiter Inhibitor verwendet wird, wobei die Anfangsdicke einer Bramme für das kornorientierte Elektrostahlblech im Bereich zwischen etwa 30 mm und 70 mm liegt und primäre Rekristallisationskörner nach Abschluß des Entkohlungsglühens einen mittleren Korndurchmesser von mindestens 7 µm und höchstens 15 µm haben.

4. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs nach Anspruch 3 mit den folgenden Schritten: auf eine Temperatur über 1050 °C und unter 1350 °C erfolgendes Nachwärmen einer Bramme mit einer Anfangsdicke von etwa 30 bis 70 mm, die in Gew.-% aufweist: 0,025 bis 0,10 % C, 2,5 bis 4,0 % Si, 0,010 bis 0,035 % säurelösliches Al, 0,0030 bis 0,010 % N, Seq = (S + 0,406 Se) von 0,008 bis 0,05 %, 0,02 bis 0,20 % Mn, optional mindestens einen Bestandteil, ausgewählt aus 0,02 bis 0,30 % Sn, Sb und/oder P, 0,01 bis 0,30 % Cu, 0,02 bis 0,30 % Cr, 0,03 bis 0,30 % Ni und 0,008 bis 0,3 % Mo und/oder Cd und als Rest Eisen sowie unvermeidliche Verunreinigungen, Warmwalzen der Bramme zu einem warmgewalzten Band, optionales Glühen des warmgewalzten Bands, einmaliges Kaltwalzen oder zwei- oder mehrmaliges Kaltwalzen des warmgewalzten Bands mit Zwischenglühen, um eine Fertigblechdicke zu bilden, Entkohlungsglühen des kaltgewalzten Blechs, Beschichten des Blechs mit einem sich hauptsächlich aus MgO zusammensetzenden Glühseparator und abschließendes Fertigglühen, wobei das Verfahren **dadurch gekennzeichnet ist, daß** es ferner den Schritt des Nitrierens des Stahlblechs nach dem Entkohlungsglühen und bis zum Beginn der sekundären Rekristallisation aufweist und primäre Rekristallisationskörner nach Abschluß des Entkohlungsglühens einen mittleren Korndurchmesser von mindestens 7 μm und höchstens 15 μm haben.

5. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach einem der Ansprüche 1 bis 4, wobei der Schritt des Nitrierens des Stahlblechs nach Entkohlungsglühen an einem laufenden Band in einer Mischgasatmosphäre durchgeführt wird, die Wasserstoff, Stickstoff und Ammoniak aufweist, um die Menge von Stickstoff im Stahlblech um 0,001 bis 0,020 Gew.-% zu erhöhen.

6. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach einem der Ansprüche 1 bis 5, wobei die Erwärmung beim Entkohlungsglühen mit einer Erwärmungsgeschwindigkeit von mindestens 100 °C/s vom Beginn des Temperaturanstiegs auf 650 bis 950 °C durchgeführt wird.

7. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach einem der Ansprüche 1 bis 6, wobei die Bandtemperatur auf Temperaturen in den folgenden Bereichen beim Warmwalzen eingestellt wird:

$$850 + 2500 \times Seq + 400 \times Mn \leq FOT\ (^\circ C) \leq 1100 + 3000 \times Seq + 800 \times Mn \leq 1350\ ^\circ C,$$

wobei FOT: Anfangstemperatur des Fertigwarmwalzens (°C),

$$800 + 2500 \times Seq + 400 \times Mn \leq FT\ (^\circ C) \leq 1050 + 3000 \times Seq + 800 \times Mn \leq 1350\ ^\circ C,$$

wobei FT: Abschlußtemperatur des Fertigwarmwalzens (°C).

8. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach einem der Ansprüche 1 bis 7, wobei die Glühbedingungen für das warmgewalzte Band auf eine maximale Temperatur von 950 bis 1150 °C und eine Glühdauer von mindestens 30 Sekunden und höchstens 600 Sekunden eingestellt sind.

9. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach einem der Ansprüche 1 bis 8, wobei das Kaltwalzen mit einem Dickenabnahmeverhältnis beim Fertigkaltwalzen von 80 bis 92 % durchgeführt wird.

10. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach einem der Ansprüche 1 bis 9, wobei das Band im Temperaturbereich von 100 bis 300 °C für mindestens 1 min während mindestens eines Fertigkaltwalzdurchgangs des Kaltwalzens gehalten wird.

11. Verfahren zur Herstellung eines kornorientierten Elektrostahlblechs mit ausgezeichneten magnetischen Kennwerten nach Anspruch 1 oder 3, wobei mindestens ein Bestandteil, ausgewählt aus 0,02 bis 0,30 % Sn, Sb und/ oder P, 0,01 bis 0,30 % Cu, 0,02 bis 0,30 % Cr, 0,03 bis 0,30 % Ni und 0,008 bis 0,3 % Mo und/oder Cd, ferner als Komponente der Bramme vorgesehen ist.

Revendications

1. Procédé de production d'une tôle d'acier électrique à grains orientés contenant de 2,5 à 4,0 % de Si, dans lequel au moins un élément choisi parmi les sulfures et des séléniures est utilisé en tant que premier inhibiteur et au moins un nitrure formé par nitruration jusqu'au début de la recristallisation secondaire suivant le recuit de décarburation est utilisé en tant que second inhibiteur, dans lequel la température de chauffage d'ébauche avant le laminage à chaud est comprise entre 1260°C et 1350°C et les grains de recristallisation primaire après l'achèvement du recuit de décarburation ont un diamètre moyen de grain non inférieur à 7 µm et non supérieur à 15 µm.

2. Procédé de production d'une tôle d'acier électrique à grains orientés selon la revendication 1, comprenant les étapes de réchauffage à une température dans la plage de 1260°C à 1350°C d'une ébauche comprenant, en % en poids, de 0,025 à 0,10 % de C, de 2,5 à 4,0 % de Si, de 0,010 à 0,035 % d'Al soluble dans l'acide, de 0,0030 à 0,010 % de N, Seq = (S + 0,406 Se) de 0,008 à 0,05 %, de 0,02 à 0,20 % de Mn, facultativement au moins l'un choisi parmi de 0,02 à 0,30 % d'au moins l'un parmi Sn, Sb et P, de 0,01 à 0,30 % de Cu, de 0,02 à 0,30 % de Cr, de 0,03 à 0,30 % de Ni et de 0,008 à 0,3 % d'au moins l'un parmi Mo et Cd, le complément
étant Fe et les impuretés inévitables, le laminage à chaud de l'ébauche en une bande laminée à chaud, facultativement le recuit de la bande laminée à chaud, la soumission à un laminage à froid ou à deux laminages à froid ou plus de la bande laminée à chaud avec un recuit intermédiaire pour former une épaisseur de tôle finale, le recuit de décarburation de la tôle laminée à froid, le revêtement de la tôle avec un séparateur de recuit composé principalement de MgO et la soumission à un recuit de finition final, dans lequel le procédé est **caractérisé en ce qu'**il comprend en outre l'étape de nitruration de la tôle d'acier de la fin du recuit de décarburation au début de la recristallisation secondaire et les grains de recristallisation primaire après l'achèvement du recuit de décarburation ont un diamètre moyen de grain non inférieur à 7 µm et non supérieur à 15 µm.

3. Procédé de production d'une tôle d'acier électrique: à grains orientés contenant de 2,5 à 4,0 % de Si, dans lequel au moins un élément choisi parmi les sulfures et des séléniures est utilisé en tant que premier inhibiteur et au moins un nitrure formé par nitruration jusqu'au début de la recristallisation secondaire après recuit de décarburation est utilisé en tant que second inhibiteur, dans lequel l'épaisseur initiale d'une ébauche pour la tôle d'acier électrique à grains orientés est dans la plage d'environ 30 mm à 70 mm et les grains de recristallisation primaire après l'achèvement du recuit de décarburation ont un diamètre de grain moyen non inférieur à 7 µm et non supérieur à 15 µm.

4. Procédé de production d'une tôle d'acier électrique à grains orientés selon la revendication 3, comprenant les étapes de réchauffage à une température supérieure à 1050°C et inférieure à 1350°C d'une ébauche formant une épaisseur initiale d'environ 30 à 70 mm comprenant, en % en poids, de 0,025 à 0,10 % de C, de 2,5 à 4,0 % de Si, de 0,010 à 0,035 % d'Al soluble dans l'acide, de 0,0030 à 0,010 % de N, Seq = (S + 0,406 Se)
de 0,008 à 0,05 %, de 0,02 à 0,20 % de Mn, facultativement au moins l'un choisi parmi de 0,02 à 0,30 % d'au moins l'un parmi Sn, Sb et P, de 0,01 à 0,30 % de Cu, de 0,02 à 0,30 % de Cr, de 0,03 à 0,30 % de Ni et de 0,008 à 0,3 % d'au moins l'un parmi Mo et Cd et le complément
étant Fe et les impuretés inévitables, le laminage à chaud de l'ébauche en une bande laminée à chaud, facultativement le recuit de la bande laminée à chaud, la soumission à un laminage à froid ou à deux laminages à froid ou plus de la bande laminée à chaud avec un recuit intermédiaire pour former une épaisseur de tôle finale, le recuit de décarburation de la tôle laminée à froid, le revêtement de la tôle avec un séparateur de recuit composé principalement de MgO et la soumission à un recuit de finition final, dans lequel le procédé est **caractérisé en ce qu'**il comprend en outre l'étape de nitruration de la tôle d'acier de la fin du recuit de décarburation au début de la recristallisation secondaire et les grains de recristallisation primaire après l'exécution du recuit de décarburation ont un diamètre de grain moyen non inférieur à 7 µm et non supérieur à 15 µm.

5. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon l'une quelconque des revendications 1 à 4, dans lequel l'étape de nitruration de la tôle d'acier après recuit de décarburation est effectuée sur une bande défilante dans une atmosphère de gaz mixte contenant de l'hydrogène, de l'azote et de l'ammoniac pour augmenter la quantité d'azote dans la tôle d'acier de 0,001 à 0,020 % en poids.

6. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon l'une quelconque des revendications 1 à 5, dans lequel le chauffage durant le recuit de décarburation est conduit à une vitesse de chauffage non inférieure à 100°C/s à partir du début de la montée en température jusqu'à 650 à 950°C.

7. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon l'une quelconque des revendications 1 à 6, dans lequel la température de la bande est ajustée à des températures dans les plages suivantes durant le laminage à chaud :

$$850 + 2500 \times Seq + 400 \times Mn \leq FOT(°C) \leq 1100 + 3000 \times Seq + 800 \times Mn \leq 1350°C,$$

où
FOT : température de début du laminage à chaud de finition (°C),

$$850 + 2500 \times Seq + 400 \times Mn \leq FT(°C) \leq 1050 + 3000 \times Seq + 800 \times Mn \leq 1350°C$$

où FT : température de fin du laminage à chaud de finition (°C).

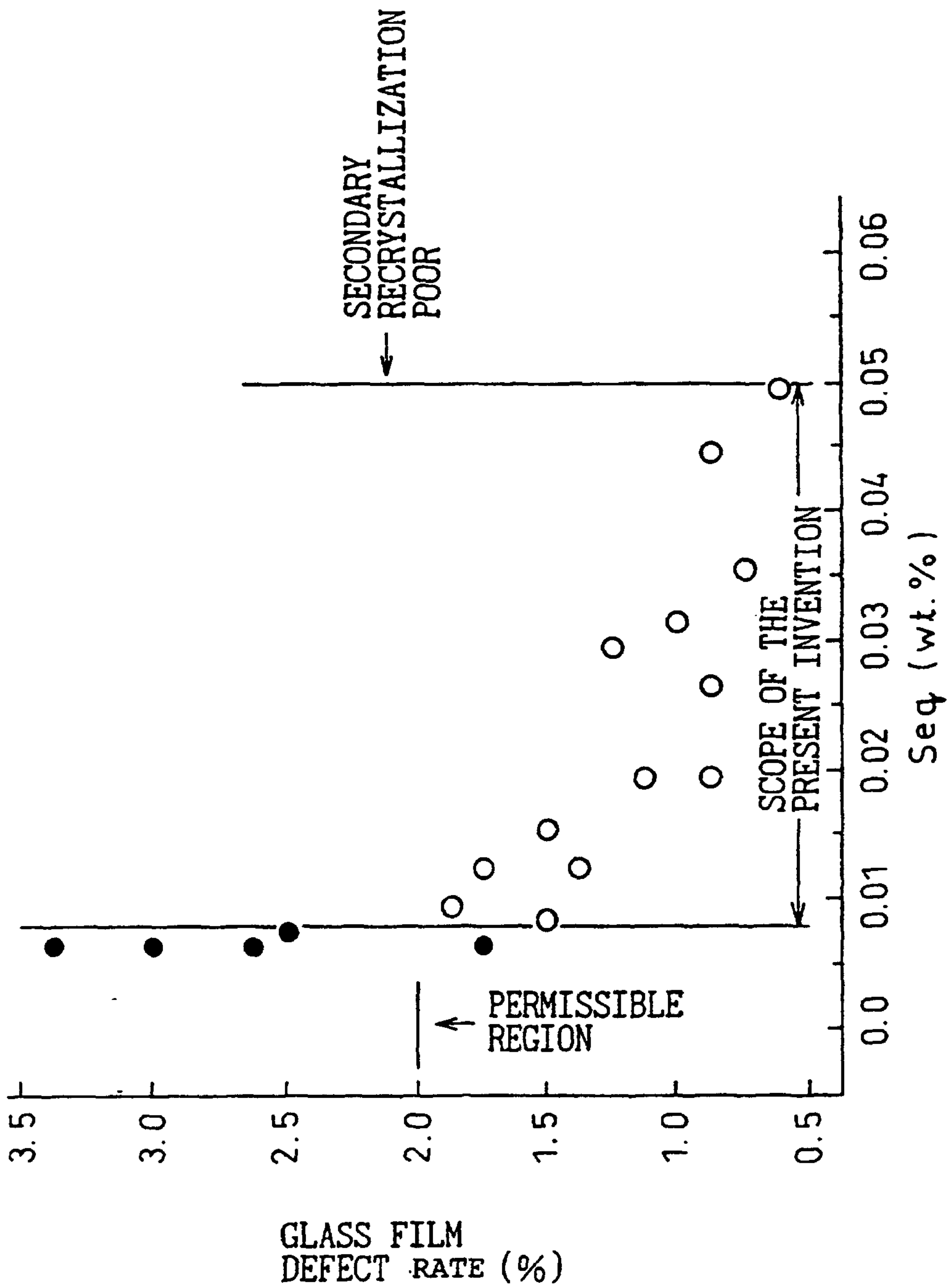
8. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon l'une quelconque des revendications 1 à 7, dans lequel les conditions de recuit de bande laminée à chaud sont ajustées à une température maximale de 950 à 1150°C et une durée de recuit non inférieure à 30 secondes et non supérieure à 600 secondes.

9. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon l'une quelconque des revendications 1 à 8; dans lequel le laminage à froid est conduit à un rapport de réduction de laminage à froid final de 80 à 92 %.

10. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon l'une quelconque des revendications 1 à 9, dans lequel la bande est maintenue dans la plage de température de 100 à 300°C pendant au moins 1 min durant au moins une passe de laminage à froid finale du laminage à froid.

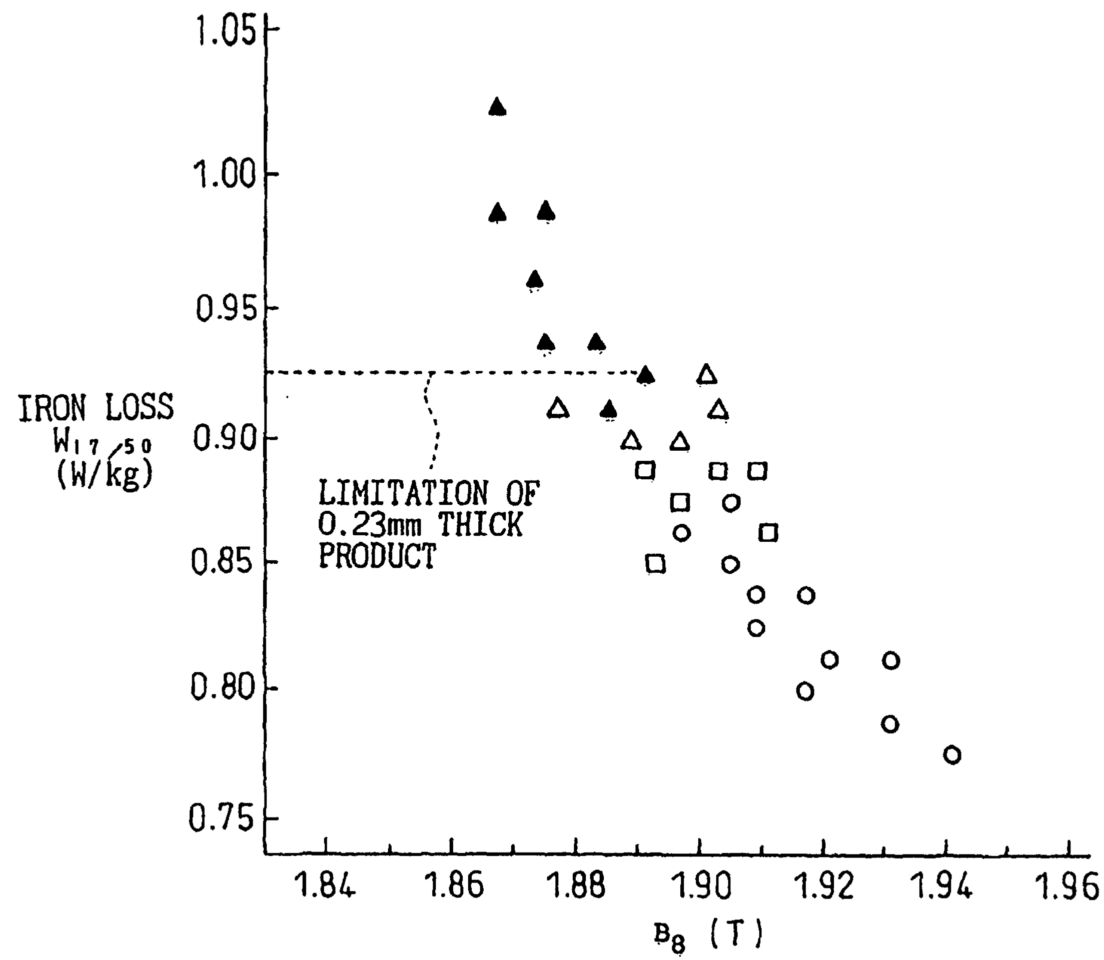
11. Procédé de production d'une tôle d'acier électrique à grains orientés excellente en terme de caractéristiques magnétiques selon la revendication 1 ou 3, dans lequel au moins l'un choisi parmi de 0,02 à 0,30 % d'au moins l'un parmi Sn, Sb et P, de 0,01 à 0,30 % de Cu, de 0,02 à 0,30 % de Cr, de 0,03 à 0,30 % de Ni et de 0,008 à 0,3 % d'au moins l'un parmi Mo et Cd est également inclus en tant que composant de l'ébauche.

Fig.1


SECONDARY RECRYSTALLIZATION POOR
SCOPE OF THE PRESENT INVENTION
PERMISSIBLE REGION
Seq (wt.%)
0.0
0.01
0.02
0.03
0.04
0.05
0.06
3.5
3.0
2.5
2.0
1.5
1.0
0.5
GLASS FILM DEFECT RATE (%)

	ATMOSPHERE DURING HEATING STAGE OF HIGH TEMPERATURE ANNEALING			
	① H_2=75%, N_2=25%	② H_2=50%, N_2=50%	③ H_2=25%, N_2=75%	④ H_2=10%, N_2=90%
WITH NITRIDING	○		□	△
WITHOUT NITRIDING	SECONDARY RECRYSTALLIZATION POOR			▲

Fig.2



	ATMOSPHERE DURING HEATING STAGE OF HIGH TEMPERATURE ANNEALING			
	① H_2=75%, N_2=25%	② H_2=50%, N_2=50%	③ H_2=25%, N_2=75%	④ H_2=10%, N_2=90%
WITH NITRIDING	○		□	△
WITHOUT NITRIDING	SECONDARY RECRYSTALLIZATION POOR		■	▲

Fig.3

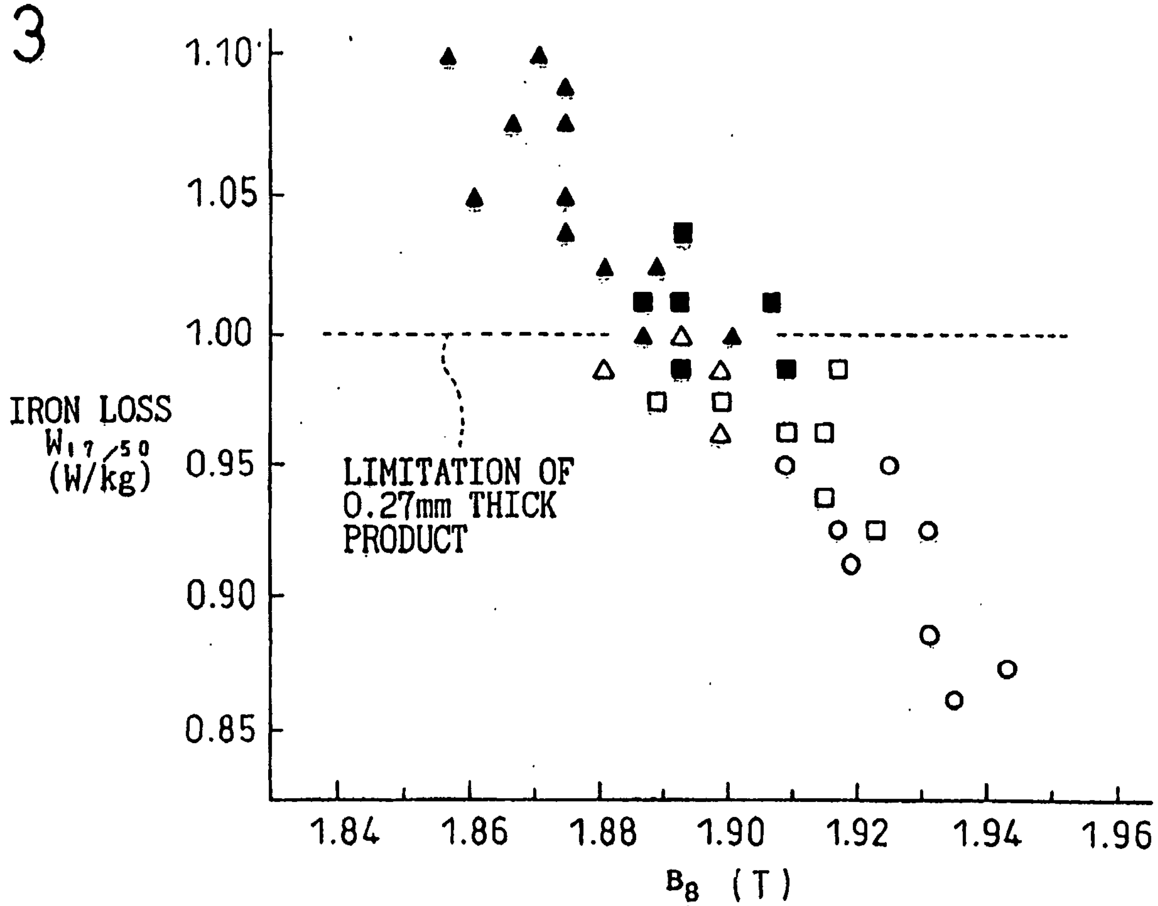
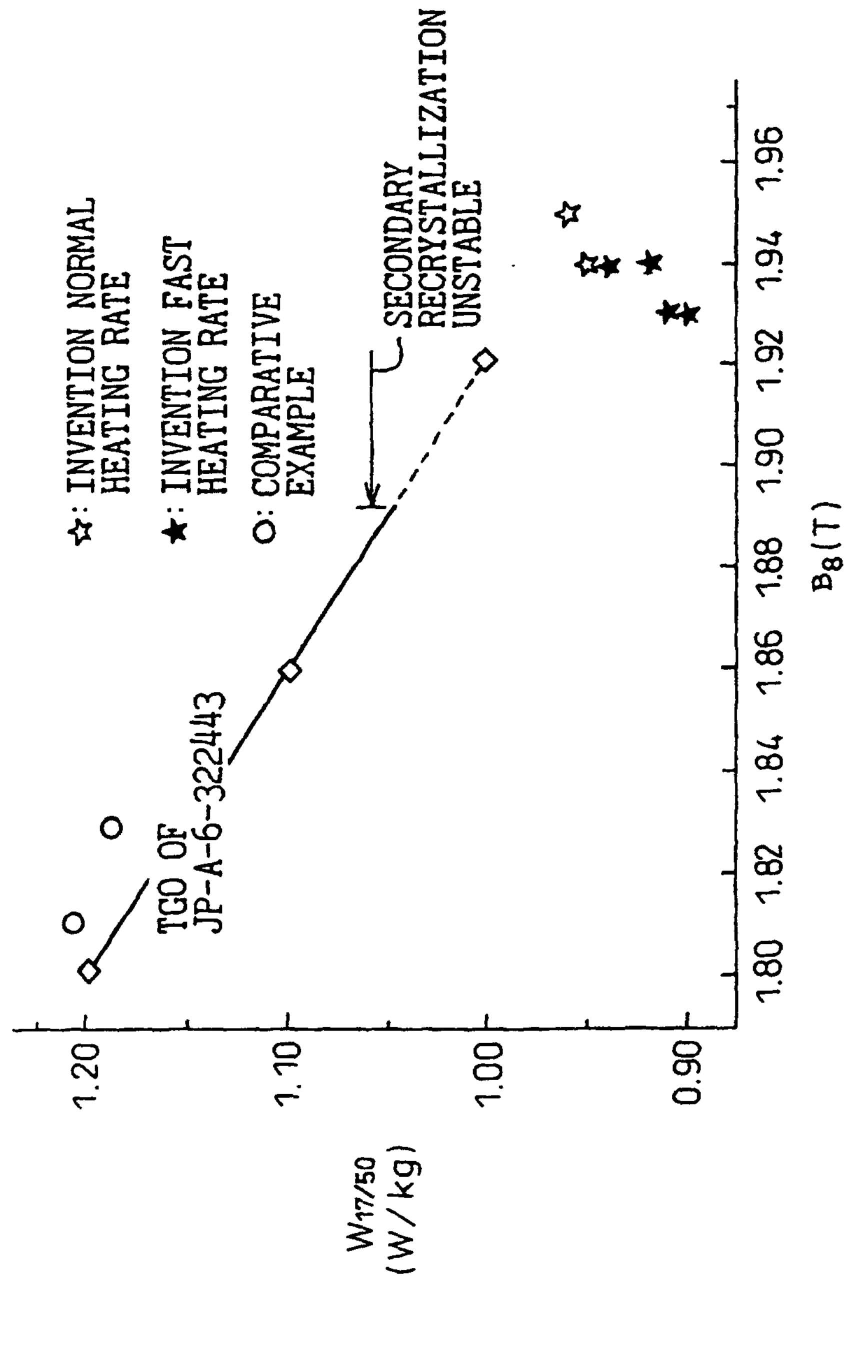


Fig.4


☆: INVENTION NORMAL HEATING RATE
★: INVENTION FAST HEATING RATE
○: COMPARATIVE EXAMPLE
SECONDARY RECRYSTALLIZATION UNSTABLE
TGO OF JP-A-6-322443
W17/50 (W/kg)
1.20
1.10
1.00
0.90
1.80
1.82
1.84
1.86
1.88
1.90
1.92
1.94
1.96
B8(T)